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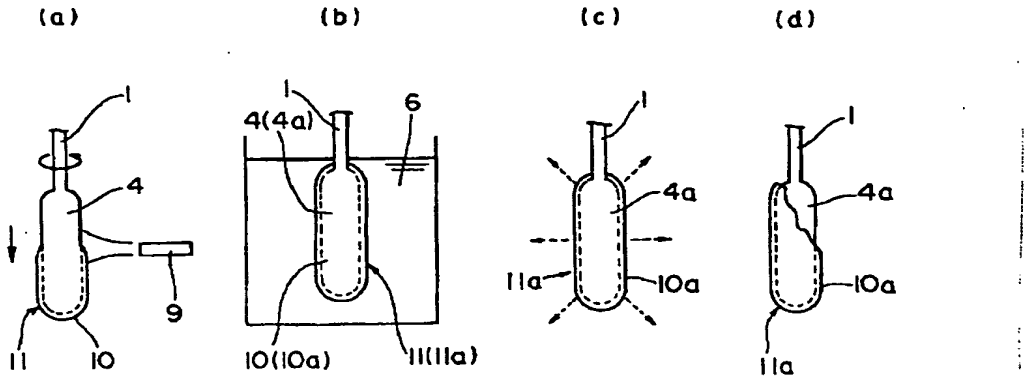
(54) [Subject of Invention]

Manufacturing method of rare earth element doped optical fiber preform

(57) [Summary]

(58) [Task] The cracking of the rare earth element doped core porous preform is prevented, and during the consolidation to transparent glass in a dehydration & sintering equipment, the breakage to the preform is prevented to enhance the production yield. [Solving Means] By depositing the protective layer 10 composed of silica fine particles to the circumference of the core porous preform 4, the protective layer attached core porous preform 11 is obtained. This protective layer attached core porous preform 11 is soaked in the alcohol solution 6 containing rare earth element chloride compound to obtain the rare earth element doped protective layer attached core porous preform 11a. This is taken out and after drying, the rare earth element doped protective layer 10a is removed from the rare earth element doped protective layer attached core porous preform 11a to taken out the rare earth element doped core porous preform 4a. This rare earth element doped core porous preform 4a is dehydrated and sintered to become transparent glass.



1...quartz target rod; 2...core burner; 3...flame; 4...core porous preform;

4a...rare earth element doped core porous preform; 5...soaking (impregnation) tub;

6...rare earth element chloride compound containing alcohol solution;

9...protective layer deposition burner; 10...protective layer;

10a...rare earth element doped protective layer; 11...protective layer attached core porous preform; 11a...rare earth element doped protective layer attached core porous preform

[Scope of the Patent Claim]

[Claim Item 1] A manufacturing method of rare earth element doped optical fiber preform having the following characteristics: The protective layer composed of silica fine particles is deposited to cover the circumference of the core porous preform formed by deposition using the axial attachment (deposition) method; the obtained protective layer attached core porous preform is soaked in an alcohol solution containing rare earth element chloride compound to obtain a rare earth element doped-protective layer attached core porous preform; the rare earth element doped protective layer attached core porous preform is taken out from the aforementioned rare earth element chloride compound alcohol solution and dried; after this, the rare earth element doped protective layer is removed from the rare earth element doped-protective layer attached core porous preform; the taken-out rare earth element doped core porous preform is dehydrated and sintered to become transparent glass.

[Claim Item 2] In the manufacturing method of the rare earth element doped optical fiber preform described in Claim Item 1, the aforementioned protective layer is set to be lower in the bulk density than the bulk density of the aforementioned core porous preform.

[Claim Item 3] In the manufacturing method of the rare earth element doped optical fiber preform described in Claim Item 1 or 2, the aforementioned protective layer is provided in the (necessary) layer number so that the aforementioned protective layer would not be stripped off during the soaking and during the drying and after the drying of the rare earth element doped core porous preform, it can be easily removed (stripped off) from the surface of the rare earth element doped core porous preform.

[Detailed Explanation of the Invention]

[0001]

[The Technical Field Belonging to the Invention] The present invention is related to a manufacturing method of rare earth element doped optical fiber preform.

[0002]

[Conventional Technology] The rare earth element doped optical fibers doped with Er (erbium), Pr (praseodymium), etc. rare earth elements in the core portion of the optical fiber are expected to be applied to optical amplifiers, optical fiber lasers, etc. In the case when a rare earth element doped optical fiber is manufactured by VAD method, etc. axial attachment (deposition) methods, so far, the doping of the rare earth element has been performed by the so called liquid soaking method (for example, cf. Patent Disclosure Bulletin No. 5-301734 [1993]).

[0003] In this liquid soaking method, first of all, as shown in Fig 2 (a), to the quartz target rod, the glass fine particles synthesized inside the flame 3 of the core burner 2 are deposited to form the core porous preform 4. Next, as shown in Fig 2 (b), the core porous preform 4 is soaked in the rare earth chloride compound containing alcohol solution 6 inside the soaking (impregnation) tub 5 to obtain the rare earth element doped core porous preform 4a. Next, the rare earth element doped core porous preform 4a taken out from the rare earth chloride compound containing alcohol solution 6 is dried at room temperature as shown in Fig 2 (c) to evaporate the alcohol content to precipitate-fix the rare earth element chloride compound inside the rare earth element doped core porous preform 4a. After this, the rare earth element doped core porous preform 4a, is, as shown in Fig 2 (d), placed into the quartz furnace core tube 8 of the dehydration-sintering

equipment 7 composed of an electric furnace and dehydrated & sintered in He, O_2 atmosphere to consolidated to transparent glass.

[0004]

[The Problem to be Solved by the Invention] However, in this kind of the conventional manufacturing method of rare earth element doped optical fiber preform, during the drying of the rare earth element doped core porous preform 4a obtained by soaking the core porous preform 4 in the rare earth element chloride compound containing alcoholic solution 6, the alcohol would be evaporated at the surface of the rare earth element doped core porous preform 4a and the surface of the rare earth element doped core porous preform 4a would be abruptly dried. Because of this, at the vicinity of the surface of the rare earth element doped core porous preform 4a, the shrinkage of the core porous preform 4a would occur markedly. But, at the inside of this core porous preform 4a, since the evaporation of the alcohol would be slow, the drying speed would be slower; thus, the shrinkage of inside of the core porous preform 4a would proceed gradually. As a result, a stress would occur between the surface vicinity of this core porous preform 4a and the inside of this core porous preform 4a. By this stress, cracking would occur on the surface of this core porous preform 4a.

[0005] The cracking occurred on the surface of the rare earth element doped core porous preform 4a would be enlarged during the consolidation to glass in the dehydration-sintering equipment 7; in some cases, the rare earth element doped core porous preform 4a would be destroyed during the manufacturing. And, the cracking occurred on the surface of the rare earth element doped core porous preform 4a would induce degradation of the surface condition to cause mismatching between the core and the clad. As a result,

in some cases the amplification performance of the rare earth element doped optical fiber obtained by fiber drawing the prepared preform would not show the expected value.

[0006] The objective of the present invention is to prevent the cracking of the rare earth element doped core porous preform and to prevent breakage to the preform during the consolidation to transparent glass in a dehydration-sintering equipment to enhance the yield.

[0007]

[The Means Used to Solve the Problem] The manufacturing method of the rare earth element doped optical fiber preform related to the invention described in Claim Item 1 is set as follows: The protective layer composed of silica fine particles is deposited to cover the circumference of the core porous preform formed by deposition using the axial attachment (deposition) method; the obtained protective layer attached core porous preform is soaked in an alcohol solution containing rare earth element chloride compound to obtain a rare earth element doped-protective layer attached core porous preform; the rare earth element doped-protective layer attached core porous preform is taken out from the aforementioned rare earth element chloride compound alcohol solution and dried; after this, the rare earth element doped protective layer is removed from the rare earth element doped-protective layer attached core porous preform; the taken-out rare earth element doped core porous preform is dehydrated and sintered to become transparent glass. As above, the circumference of the core porous preform is covered by the protective layer composed of silica fine particles; and when the obtained protective layer attached core porous preform is soaked in the rare earth element chloride compound containing alcoholic solution, the rare earth element chloride compound containing alcoholic solution

would be impregnated into the protective layer and the core porous preform (through the protective layer). When it is pulled up and dried, the alcohol would be evaporated form the surface of the rare earth element doped protective layer of the outside; thus cracking would occur to the rare earth element doped protective layer by the abrupt drying. However, the rare earth element doped core preform of the inside wrapped by the rare earth element doped protective layer would be gradual in alcohol evaporation since it is wrapped inside the rare earth element doped protective layer and the drying speed from the surface of the rare earth element doped core porous preform would be suppressed; thus, the occurrence of the cracking by the abrupt drying encountered in the rare earth element doped protective layer could be prevented. When the rare earth element doped protective layer degraded by the cracking is removed after the completion of the drying of the rare earth element doped core porous preform wrapped by the rare earth element doped protective layer, a rare earth element doped core porous preform without cracking formation can be obtained.

[0008] In the manufacturing method of the rare earth element doped optical fiber preform related to the invention described in Claim Item 2, the protective layer is set to be lower in the bulk density than the bulk density of the core porous preform; by the density difference of the two, the removal operation of the protective layer can be performed easier.

[0009] In the manufacturing method of the rare earth element doped optical fiber preform described in the invention related to Claim Item 3, it is set so that the protective layer wrapping the core porous preform would not be peeled off during the soaking and during the drying; and it is arranged in the (necessary) layer number so that after the drying of the rare earth element doped core porous preform, it can be easily removed (stripped off)

from the surface of the rare earth element doped core porous preform. By providing the protective layer as above, the protective layer would not be stripped off during the soaking and during the drying, but it can be easily stripped off (separated) from the surface of the core porous preform after the drying.

[0010]

[Implementation Mode of the Invention] Fig 1 (a)—(d) show an example of the implementation mode of the manufacturing method of the rare earth element doped optical fiber preform related to the present invention. First of all, as shown in Fig 1 (a), while the core porous preform 4 deposited onto the quartz target rod 1 is being rotated, onto the surface, the protective layer 10 composed of silica fine particles of below 0.2 g/cm^3 in bulk density synthesized by the protective layer deposition burner 9 are deposited to cover the surface to obtain the protective layer attached core porous preform 11. In this case, from the protective layer deposition burner 9, H_2, O_2, etc. combustion gases and the SiO_2 fine particles formed by hydrolysis reaction of glass raw materials are sprayed to deposit 1 to 2 layers of the protective layer 10 of bulk density below 0.2 g/cm^3. The bulk density of this protective layer 10 is lower than the bulk density (0.2—0.3 g/cm^3) of the core porous preform 4.

[0011] Next, as shown in Fig 1 (b), the protective layer attached core porous preform 11 is soaked in the rare earth chloride compound containing alcohol solution 6 inside the soaking (impregnation) tub 5. By this, the rare earth chloride compound containing alcohol solution 6 would be impregnated into the protective layer 10 and further be impregnated into the porous preform 4 by passing through the protective layer 10: the protective layer 10 would become the rare earth element doped protective layer 10a and

the core porous preform 4 would become the rare earth element doped core porous preform 4a; thus from the protective layer attached core porous preform 11, the rare earth element doped protective layer attached core porous preform 11a would be obtained. Next, the rare earth element doped protective layer attached core porous preform 11a taken out from the rare earth chloride compound containing alcohol solution 6 is dried at room temperature as shown in Fig 1 (c) to evaporate the alcohol content. By this, the rare earth element chloride compound inside the rare earth element doped protective layer attached core porous preform 11a are precipitated-fixed. Next, when the rare earth element doped protective layer attached core porous preform 11a is completely dried, the rare earth element doped protective layer 10a on the surface of the rare earth element doped protective layer attached core porous preform 11a can be separated by the knife tip of cutter-knife, etc. to take out the inside rare earth element doped core porous preform 4a, as shown in Fig 1 (d). In the taking-out of this rare earth doped core porous preform 4a, since the surface of the rare earth element doped protective layer 10a has been degraded and cracked by the drying, it can be easily stripped-separated by the knife tip of cutter-knife, etc. In doing this, the rare earth element doped core porous preform 4a inside the rare earth element doped protective layer 10a can be taken out without damaging.

[0012] The protective layer is arranged in the (necessary) layer number so that it would not be stripped off during the soaking in the rare earth chloride compound containing alcohol solution 6 inside the soaking (impregnation) tub 5 or during the drying for evaporating the alcohol after the soaking in the alcohol solution 6 and also that after the drying of the rare earth element doped core porous preform, it can be easily removed

(stripped off) from the surface of the rare earth element doped core porous preform 4a. After this, the rare earth element doped core porous preform 4a is, as shown in Fig 2 (d), placed into the quartz furnace core tube 8 of the dehydration-sintering equipment 7 composed of an electric furnace and dehydrated & sintered to consolidated to transparent glass. By the manufacturing as above, the rare earth element doped core porous preform 4a superior in surface condition can be obtained.

[0013]

[Implementation Example]

--Synthesis Conditions of the Core Porous Preform 11a

$SiCl_4$ 162 SCCM, Carrier O_2 200 SCCM; $GeCl_4$ 12.2 SCCM, Carrier O_2 200 SCCM; FG-O_2 1.2 SLM, FG-CH_4 1.2 SLM; IS 6.1 SLM, OS 200 SCCM (end burner FG-O_2 2.8 SLM, FG-CH_4 3.0 SLM)

--Protective Layer Condition

$SiCl_4$ 1.4 SLM, Carrier O_2 0.9 SLM; FG-O_2 1.4 SLM, FG-CH_4 2.0 SLM; IS 1.0 SLM, OS 1.24 SLM (end burner FG-O_2 5.5 SLM, FG-CH_4 6.0 SLM)

--Turn Number: 2 (round trip)

--Property of the Core Porous Preform

$\rho = 0.364$ g/cm^3

--Soaking Condition

Solution alcohol, Solution volume 1000 ml, Soaking time 5 H

--Drying Condition

Atmosphere air, Drying Time 50 H

In this test preparation, the objective was to prevent cracking of the core porous preform during the soaking in the alcohol solution and during the drying; thus no dopants such as $ErCl_4$ and/or $AlCl_4$, ($ErCl_4$ and/or $AlCl_4$,; obvious misprints of $ErCl_3$ and/or $AlCl_3$) etc. were not added

[0014]

[Effect of the Invention] According to the manufacturing method of the rare earth element doped optical fiber preform related to the present invention, the circumference of the core porous preform is covered by a protective layer composed of silica fine particles, the obtained protective layer attached core porous preform is soaked in an alcohol solution containing rare earth element chloride compound, and this is then pulled up and dried. By this, cracking would occur on the outside rare earth element doped protective layer by the abrupt drying; however, the drying speed of the inside rare earth doped core porous preform would be suppressed by the presence of the rare earth element doped protective layer so that the occurrence of cracking to the core porous preform can be prevented. Because of this, the breakage to the preform during the consolidation to transparent glass in a dehydration-sintering equipment can be prevented to enhance the production yield.

[0015] And, based on the present invention, the rare earth element chloride compound can be doped while the surface of the core porous preform is maintained in smooth condition; as a result, a rare earth element doped optical fiber possessing superior amplification performance can be manufactured.

[0016] And, since the protective layer composed of silica fine particles would crack by the drying after the rare earth element chloride compound containing alcohol solution

soaking; thus after the drying, the protective layer can be easily removed from surface of the rare earth element doped core porous preform.

[0017] Furthermore, by the protective layer composed of silica fine particles, when the core porous preform wrapped with the protective layer is soaked in the rare earth element chloride compound containing alcohol solution, it can be protected from doping of foreign materials.

[0018] Moreover, by making the bulk density of the protective layer lower than the bulk density of the core porous preform, due to the density difference between the two, the protective layer removal operation after the drying can be performed much easier.

[0019] Further, in setting the protective layer to a suitable layer number so that the protective layer would not be stripped off during the soaking in the rare earth element chloride compound containing alcohol solution and during the drying of the rare earth element doped protective layer attached core porous preform 11a and after the drying of the rare earth element doped protective layer attached core porous preform, the protective layer can be easily stripped off from the surface of the core porous preform, the protective layer would not be stripped off during the soaking in the rare earth element chloride compound containing alcohol solution and during the drying of the rare earth element doped protective layer attached core porous preform 11a, but it can be easily stripped off (separated) from the surface of the core porous preform.

[Brief Explanation of Figures]

Fig 1 (a)—(d) are the process diagrams of an example of the implementation mode in the manufacturing method of the rare earth element doped optical fiber preform related to the present invention.

Fig 2 is the process diagrams of the manufacturing method of the conventional rare earth element doped optical fiber preform.

[Illustration of Symbols]

1.....quartz target rod

2.....core burner

3.....flame

4.....core porous preform

4a....rare earth element doped core porous preform

5.....soaking (impregnation) tub

6.....rare earth element chloride compound containing alcohol solution

7.....dehydration and sintering equipment

8.....quartz furnace core tube

9.....protective layer deposition burner

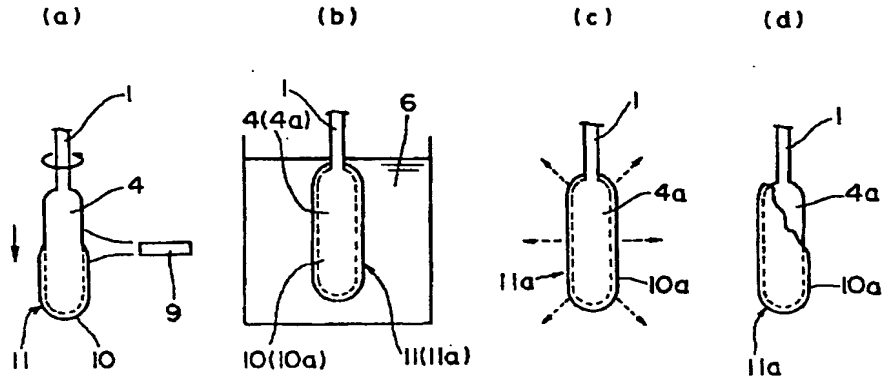
10....protective layer

10a...rare earth element doped protective layer

11....protective layer attached core porous preform

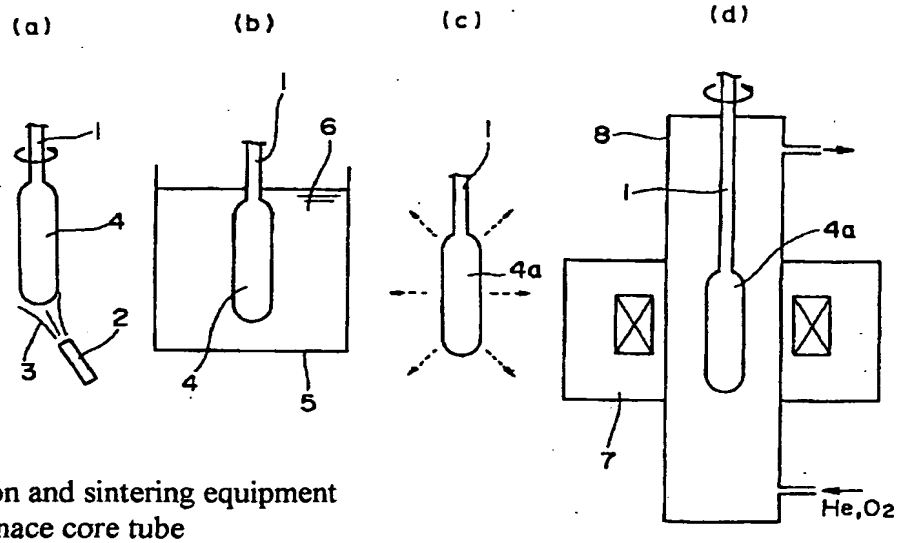
11a...rare earth element doped protective layer attached core porous preform

Fig 1



1...quartz target rod; 2...core burner; 4...flame; 4...core porous preform; 4a...rare earth element doped core porous preform; 5...soaking (impregnation) tub; 6...rare earth element chloride compound containing alcohol solution; 9...protective layer deposition burner; 10...protective layer; 10a...rare earth element doped protective layer; 11...protective layer attached core porous preform; 11a...rare earth element doped protective layer attached core porous preform

Fig 2



7.....dehydration and sintering equipment
8.....quartz furnace core tube

An amendment was submitted on May 20, 1996 by which the section [0007] [The Means Used to Solve the Problem] was amended.

(This amendment was incorporated in the translation.)